

DEVELOPMENT OF HEAT-SEALABLE SEWING THREAD

PHASE I

BY
R. WHITE
C. MOWREY
J. LABIS

SPRINGBORN LABORATORIES, INC.
ENFIELD, CONNECTICUT 06082-4899

MAY 1988
FINAL REPORT FOR THE PERIOD
AUGUST 1986 TO APRIL 1987

19990105 064
490

APPROVED FOR PUBLIC RELEASE;
DISTRIBUTION UNLIMITED.

UNITED STATES ARMY NATICK
RESEARCH, DEVELOPMENT AND ENGINEERING CENTER
NATICK, MASSACHUSETTS 01760-5000

INDIVIDUAL PROTECTION DIRECTORATE

DISCLAIMERS

The findings contained in this report are not to be construed as an official Department of the Army position unless so designated by other authorized documents.

Citation of trade names in this report does not constitute an official endorsement or approval of the use of such items.

DESTRUCTION NOTICE

For Classified Documents:

Follow the procedures in DoD 5200.22-M, Industrial Security Manual, Section II-19 or DoD 5200.1-R, Information Security Program Regulation, Chapter IX.

For Unclassified/Limited Distribution Documents:

Destroy by any method that prevents disclosure of contents or reconstruction of the document.

Unclassified

SECURITY CLASSIFICATION OF THIS PAGE

Form Approved
OMB No. 0704-0188
Exp. Date: Jun 30, 1986

REPORT DOCUMENTATION PAGE

1a. REPORT SECURITY CLASSIFICATION Unclassified		1b. RESTRICTIVE MARKINGS	
2a. SECURITY CLASSIFICATION AUTHORITY		3. DISTRIBUTION/AVAILABILITY OF REPORT Approved for public release; distribution unlimited.	
2b. DECLASSIFICATION/DOWNGRADING SCHEDULE			
4. PERFORMING ORGANIZATION REPORT NUMBER(S) Project 6074.14		5. MONITORING ORGANIZATION REPORT NUMBER(S) NATICK/TR-87/046	
6a. NAME OF PERFORMING ORGANIZATION Springborn Laboratories, Inc.	6b. OFFICE SYMBOL (If applicable)	7a. NAME OF MONITORING ORGANIZATION U.S. Army Natick RD&E Center	
6c. ADDRESS (City, State, and ZIP Code) Enfield, Connecticut 06082-4899		7b. ADDRESS (City, State, and ZIP Code) Kansas Street Natick, Massachusetts 01760-5001	
8a. NAME OF FUNDING/Sponsoring Organization	8b. OFFICE SYMBOL (If applicable)	9. PROCUREMENT INSTRUMENT IDENTIFICATION NUMBER DAAK60-86-C-0089	
8c. ADDRESS (City, State, and ZIP Code)		10. SOURCE OF FUNDING NUMBERS	
		PROGRAM ELEMENT NO. SBIR	PROJECT NO. A86-135
		TASK NO.	WORK UNIT ACCESSION NO.
11. TITLE (Include Security Classification) Development of Heat-Sealable Sewing Thread - Phase I			
12. PERSONAL AUTHOR(S) R. White, C. Mowrey, J. Labis			
13a. TYPE OF REPORT Final	13b. TIME COVERED FROM 8/86 TO 4/87	14. DATE OF REPORT (Year, Month, Day) 1987 May	15. PAGE COUNT 45
16. SUPPLEMENTARY NOTATION			
17. COSATI CODES		18. SUBJECT TERMS (Continue on reverse if necessary and identify by block number)	
FIELD	GROUP	SUB-GROUP	Thread Hot Melt Tape Hydrostatic Resistance Fabrics Coatings Peel Strength Sealed Systems Adhesives
19. ABSTRACT (Continue on reverse if necessary and identify by block number)			
<p>Developmental CP fabrics contain a waterproof breathable membrane (Gore-Tex)® barrier which is laminated to the underside of an outer shell fabric. This barrier provides liquid water resistance and some degree of agent resistance. During sewing this barrier becomes perforated, inviting penetration by liquid and vaporous agents, thereby posing a severe hazard to the soldier. This problem is presently handled by using a hot melt tape to seal the thread bundles and holes generated by sewing.</p> <p>The purpose of this program was to screen and evaluate hot melt coatings for thread which could heat-seal the holes made during sewing.</p> <p>Commercially available hot melt coatings investigated included: polyesters, polyamides, polyurethanes, vinyls, and ethylene copolymers.</p>			
20. DISTRIBUTION/AVAILABILITY OF ABSTRACT <input checked="" type="checkbox"/> UNCLASSIFIED/UNLIMITED <input type="checkbox"/> SAME AS RPT. <input type="checkbox"/> DTIC USERS		21. ABSTRACT SECURITY CLASSIFICATION Unclassified	
22a. NAME OF RESPONSIBLE INDIVIDUAL S. Szczesuil		22b. TELEPHONE (Include Area Code) (617) 651-4695	22c. OFFICE SYMBOL STRNC-ITFT

Unclassified

19. Abstract - continued

Quantitative peel strength data for the hot melts versus polyester, nylon and Gore-Tex® were developed. The best hot melts were coated onto nylon thread, which was then sewn into nylon Taslan*/Gore-Tex/ tricot knit cloth. The sewn cloth samples were tested for hydrostatic resistance.

Based on the results of the hydrostatic resistance tests, the adhesive chosen for pilot plant coating and machine sewing was Michem® 4990, an ethylene/acrylic acid copolymer based material supplied by Michelman/Dow Chemical Company.

In summary, this Phase I effort demonstrated the concept of coating thread with a hot melt resin and briefly heating the sewn seam of an outer shell fabric to effect a seal, thereby preventing hydrostatic leakage. Several hot melts were identified as promising for bonding nylon to Gore-Tex. However, hydrostatic testing on sewn and sealed seams indicated that Michem 4990 had the most immediate promise. Therefore, the Michem resin was used for larger scale thread coating and evaluation.

* DuPont manufacturing process using looped yarn, two different yarn tensions.

SUMMARY

Commercial material suppliers were contacted for their recommendations on specific grades of hot melt resins (preferably low melting) for bonding nylon and/or polyester to Gore-Tex.® Sample laminates were prepared consisting of the substrate (nylon, polyester, Gore-Tex) and the adhesive. The peel strength of the adhesive versus substrate was evaluated dry and, if promising, immersed in water for 48 hours. Thread was coated on a laboratory scale with the most promising adhesives. Hand sewn samples were heated in an oven to melt the adhesive and screened for hydrostatic resistance with a laboratory pressure filter. The five most promising of these were evaluated for hydrostatic resistance as per Federal Standard 191A, TM 5512.

The adhesive chosen for pilot plant coating and machine sewing was the Michem 4990 based on the results of the hydrostatic resistance test.

A pilot plant scale coating apparatus was constructed to coat large amounts of thread for machine sewing. Fluorinert (Fluorocarbon - 3M) was used as a lubricant to aid in the sewing process. The sewability (small sewing machine) of the triple-coated Michem 4990 thread with the use of the lubricant was demonstrated and found to be very comparable to uncoated thread.

The results of the hydrostatic resistance testing were lower for samples sewn and sealed with the pilot plant-coated green thread than for those using the coated white thread, 12 to 14 psig versus an average 57 psig. The green thread was coated as received while the white thread was solvent washed prior to coating. These results suggest that a solvent wash may be required before coating the thread in order to obtain satisfactory hydrostatic resistance in the final sewn and sealed seams.

The following conclusions can be drawn from this Phase I program:

- * It is feasible to coat nylon thread with a hot melt resin, sew a shell fabric with the resulting thread, heat the seam briefly to effect a seal, and thereby prevent hydrostatic leakage in the area of the sewn and sealed seam.
- * Of the hot melt resins, Michem 4990, a water dispersion of ethylene-acrylic acid copolymer, provided sewn and sealed fabric laminates with the highest and most reproducible hydrostatic resistance.
- * Selected polyamide, urethane, ethylene-vinyl acetate and ethylene-acid hot melt resins also appear promising as nylon thread coatings for this application.
- * Sewability was demonstrated using nylon thread coated with the preferred resin and a small portable sewing machine.

PREFACE

The effort documented in this report was conducted by the following Springborn Laboratories (S/L) personnel:

Roy A. White	Project Manager
Carol A. Mowrey	Project Chemist
John J. Labis	Senior Technician

Natick personnel:	Thomas G. Jacques	Administration
	Steve Szczesuil	Contracting Officer
		Project Officer

The work described herein was conducted under contract number DAAK60-86-C-0089, S/L project number 6074.14 and carried out during the period August 1986 to April 1987.

The author wishes to acknowledge the many helpful contributions of Mr. Steve Szczesuil, Project Officer, Natick Research, Development and Engineering Center.

CONTENTS

	<u>Page</u>
SUMMARY	iii
PREFACE	iv
LIST OF FIGURES.....	vi
LIST OF TABLES.....	vi
I. INTRODUCTION	1
II. DEVELOPMENTAL	2
A. Materials	2
B. Initial Screening of Hot Melt Coatings	3
1. Peel Strength Adhesion of the Hot Melt Coating to Nylon and Polyester.....	3
2. Peel Strength Adhesion of the Hot Melt Coating to Gore-Tex.....	3
C. Coated Thread - Laboratory Scale	7
1. Adhesive Formulations	7
2. Initial Thread Coating	7
3. Initial Hand Sewing	10
D. Evaluation of Volatile Yarn Lubricants.....	18
E. Hydrostatic Pressure Resistance	18
III. DISCUSSION OF RESULTS	22
A. Initial Screening	22
1. Peel Strength Adhesion of the Hot Melt Coatings to Nylon and Polyester	22
2. Peel Strength Adhesion of the Hot Melt Coating to Gore-Tex	23
3. Evaluation of Coated Thread	23
4. Pilot Plant-Coated Thread/Machine Sewing	25
5. Volatile Yarn Lubricants	25
B. Most Promising Candidate	26
1. Thread Coating	26
2. Machine Sewing	26
3. Machine Sewn Samples	28
IV. CONCLUSIONS	29
V. RECOMMENDATIONS	30
LIST OF REFERENCES	33
APPENDIX: Hot Melt Materials.....	35

LIST OF FIGURES

	<u>Page</u>
FIGURE 1 Test Specimen, Three Stitch, Single Thread.....	11
FIGURE 2 Test Specimen, Three Stitch, Double Thread	11
FIGURE 3 Test Specimen, Three Stitch, Quadruple Thread.....	11
FIGURE 4 Test Specimen, Sewing Machine Needle, Three Stitch, Double Thread.....	12
FIGURE 5 Sewing Machine Stitch, Loop Within Needle Hole (Type 301).....	12
FIGURE 6 Pilot Plant Thread Coater.....	27

LIST OF TABLES

TABLE 1 Preliminary Candidate Screening - Peel Strength (1/2" Width Sample).....	4
TABLE 2 Gore-Tex Laminations - Peel Strength (1/2" Width Samples).....	8
TABLE 3 Thread Coating Formulations	9
TABLE 4 Preliminary Screening - Pressure Filter Test (Single Thread)	13
TABLE 5 Pressure Filter Test - Double Thread	14
TABLE 6 Pressure Filter Test - Double Thread	15
TABLE 7 Pressure Filter Test - Quadruple Thread	16
TABLE 8 Summary, Pressure Filter Tests	17
TABLE 9 Friction Test - Adhesive vs Metal	19
TABLE 10 Michem Prime 4990 Friction Test w/Various Lubricants	20
TABLE 11 Hydrostatic Pressure Resistance	21

DEVELOPMENT OF HEAT-SEALABLE SEWING THREAD

I. INTRODUCTION

Developmental CP fabrics contain a waterproof breathable membrane (Gore-Tex) ⁽¹⁾ barrier that is laminated to the underside of an outer shell fabric. This barrier provides liquid water resistance and some degree of agent resistance. During sewing this barrier becomes perforated, inviting penetration by liquid and vaporous agents, thereby posing a severe hazard to the soldier. This problem is presently handled by using a hot melt tape to seal both the thread bundles and holes generated by sewing.

The purpose of this program was to demonstrate the feasibility of using a hot melt thread coating to seal nylon and polyester thread to a sewn Gore-Tex/outer shell fabric laminate. It was an objective that this hot melt coating, when reheated, would fill the needle holes and thread interstices so as to provide a barrier to vapors and liquids in sewn and sealed seam areas. Specifically, the sewn and sealed seams should provide a hydrostatic resistance of at least 35 psig, a criterion established by Natick RD&E under previous contract DAAK60-84-C-0052.

This program addressed two basic areas of feasibility: (1) Is it possible to satisfactorily machine sew Gore-Tex/shell fabric laminate using a thread which has been coated with a hot melt resin? Will problems of softening resin or insufficient lubricity prevent realistic sewing speeds? and (2) Is it possible to heat the sewn seams and fuse the hot melt resin in such a way as to seal the thread bundles and surrounding needle holes? Can this be done at a temperature which will not adversely affect the cloth and thread and which will provide adequate hydrostatic resistance?

In pursuit of these objectives, a number of commercially available hot melt coatings were selected and evaluated for adhesion to nylon, polyester, and Gore-Tex, for their ability to be coated onto the thread, for surface lubricity and sewability, and for their ability to provide hydrostatic resistance when applied to a thread and used in a sewn and sealed seam. The most promising hot melt was used to coat pilot quantities of thread which were submitted to Natick RD&E for independent evaluation.

II. DEVELOPMENTAL

A. Materials

Fabric laminates, sewing thread and candidate hot melt coating resins were procured as follows:

Fabric Laminate: Natick RD&E Center provided a nylon Taslan^{*}/Gore-Tex/tricot knit fabric lammed as representative of the outer shell construction in a chemical protective garment.

Sewing Thread: Natick RD&E Center provided nylon thread, V-T-295 Type I, Class A, Size E, in both green and white and in various lengths for both laboratory investigation as well as initial pilot plant coating. For the coating of 2000 feet of thread, one spool of white nylon thread V-T-295, Type I, Class A (no sizing) was received from Coats and Clark.

Hot Melt Coatings: Candidate commercially available materials were selected and procured from vendors based on the following criteria:

- Likelihood, based on chemical structure, of providing good adhesion to nylon, polyester, and Gore-Tex.
- Good lubricity and low surface tack for sewability.
- Good water resistance.
- Low melt viscosity and good flow-out on heating for efficient sealing.
- Adaptable to production coating of thread. For reasons of environmental protection, organic solvent based coatings were avoided where possible, and water dispersion and direct hot melt coating systems were given preference.

Candidate hot melt coatings were selected from the following resin classes; polyester, polyamide, polybutylene, ethylene/acrylic or methacrylic acid copolymer, and vinylidene chloride based copolymers. A complete list of resin grades and manufacturers appears in the Appendix.

* DuPont manufacturing process using looped yarn, two different yarn tensions.

B. Initial Screening of Hot Melt Coatings

1. Peel Strength Adhesion of the Hot Melt Coating to Nylon and Polyester

In order for the hot melt coatings to perform satisfactorily, they must have adequate adhesion to the sewing thread, either nylon or polyester. To develop quantitative data on peel strength adhesion, hot melt coatings were applied to nylon and polyester and tested for "T" peel. In order to develop basic adhesion data, flat films of nylon and polyester were substituted for the thread.

Film/hot melt/film test laminates were prepared as follows:

First, free-standing sheets of the hot melt coating candidates were prepared. Most resins were compression molded into 4- to 5-mil-thick sheets at 330°F using Teflon coated press plates to facilitate release. Where the coating had been supplied as a water dispersion, the dispersion was cast onto a glass plate and the air-dried film stripped from the glass.

These hot melt films were then used to prepare nylon-hot melt-nylon and polyester-hot melt-polyester peel test laminates. The nylon and polyester films were first solvent cleaned and the laminates were prepared again by compression molding at 350°F and 39 psig. These laminates were cut into 1/2-inch-wide strips for peel testing. For convenience in reporting, the grams/inch factor was utilized.

Peel strength was determined with the peel force measured at a 90-degree angle to the plane of the sample. Results were reported as the average force in grams per unit width of test laminate. These initial peel test results appear in Table 1. During peel testing, force values generally oscillate and the range of values obtained is presented.

The most promising laminates, based on the initial peel strength data, were immersed in water at room temperature for 48 hours. The samples were then removed and, while still wet, retested for peel strength.

Based on previous experience, a peel strength of less than 200 grams/inch of width was considered inadequate for the application. Such a low adhesive strength would likely result in delamination of the hot melt coating from the sewing thread in the end-item with possible loss of hydrostatic resistance in the sewn and sealed garment.

2. Peel Strength Adhesion of the Hot Melt Coating to Gore-Tex

In order for the hot melt coatings to perform satisfactorily they must also have adequate adhesion to the Gore-Tex barrier. Again, in order to develop quantitative data on peel strength adhesion, hot melt coatings were applied to Gore-Tex films and tested for "T" peel.

TABLE 1

Preliminary Candidate Screening - Peel Strength (1/2" Width Sample)

Sample I.D. Number	Adhesive	Laminate ^{a.} Material	Adhesive Failure f.f. (g/in.) ^{b.} Range	Water Immersed at RT for 48 hrs (g/in.)
22006-1 A	Vitel 10,202	Nylon	0	h.
22006-1 B	(polyester)	Nylon ^{b.}	0	h.
22006-1 C		Mylar	0	h.
22006-2 A	Vitel 4709A	Nylon	0	h.
22006-2 B	(polyester)	Nylon ^{b.}	0	h.
22006-2 C		Mylar	2200-2400	300-1000
22006-3 A	Estane 5701	Nylon	100-230	h.
22006-3 C	(urethane)	Mylar	10-20	h.
22006-4 A	Estane 5715	Nylon	100-400	140-260
22006-4 C	(urethane)	Mylar	500-800	500-740
22006-5 A	Dow EAA 459	Nylon	250	h.
22006-5 C	(ethylene-acid)	Mylar	0	h.
22006-6 A	Surlyn 9970	Nylon	100-140	h.
22006-6 C	(ionomer)	Mylar	0	h.
22006-7 A	TPX 11-808 ^{j.}	Nylon	100-300	h.
22006-7 B	(polyamide)	Nylon	80-300	h.
22006-7 C		Mylar	0	h.
22002-A-3	Elvamide 8023 (polyamide)	Nylon ^{b.}	120-500	h.
22003-A	Elvamide 8061 ^{j.} (polyamide)	Nylon ^{b.} Mylar	60-600 0	100-440-660 (Nylon tore)
22004-3	Elvamide 8063 (polyamide)	Nylon ^{b.}	100-180	h.
22005-A-1	Bostik 7376 (polyester)	Nylon ^{b.} Mylar	40-60 0-60	h. h.

- a. Molded and press-laminated 330° F
- b. Aged less than 7 days
- c. Molding and laminating at 275° F
- d. Molding and laminating at 300° F
- e. Brittle when wet
- f. Number of experiments as shown in table
- g. Peel values generally oscillate during test
- h. Test eliminated due to substandard dry peel
- i. Grams peel/inch width
- j. Fluid melt
- k. Yields unusually fluid melt
- l. Received late in program
- m. Poor friction characteristics, not tested further

TABLE 1 - Continued

Preliminary Candidate Screening - Peel Strength (1/2" Width Sample)

Sample I.D. Number	Adhesive	Laminate ^{a.} Material	Adhesive Failure in grams/inch ^{b.} Range	Water Immersed at RT for 48 hrs (gms/in.)
22007-1	Michem 4990 Prime (ethylene-acid dispersion)	Nylon Mylar	100-850 0	300-360 h.
22007-2	Neo-Rez R960	Nylon Mylar	400-700 0	0-100 milky white appearance
22007-3	Duraflex DP 8910 (polybutylene)	Nylon ^{c.} Nylon ^{d.} Mylar ^{c.} Mylar ^{d.}	0 0 0 0	h. h. h. h.
22008-1	Nucrel 699	Nylon Nylon Mylar Mylar	18-240 300-420 0 0	h. h. h. h.
22008-2	Nucrel 925	Nylon Nylon Mylar Mylar	300-550 300-840 0 0	400-420 h. h. h.
22008-3	Hytrel 4056	Nylon Nylon Mylar Mylar	100-150 120-150 300-460 300-700	60-160 0 1000-2000 500-800
22008-4	Bostick 7155	Nylon Nylon Mylar Mylar	20-30 20-30 low (~20) low (~20)	h. h. h. h.

- a. Molded and press-laminated 330°F
- b. Aged less than 7 days
- c. Molding and laminating at 275°F
- d. Molding and laminating at 300°F
- e. Brittle when wet
- f. Number of experiments as shown in table
- g. Peel values generally oscillate during test
- h. Test eliminated due to substandard dry peel
- i. Grams peel/inch width
- j. Fluid melt
- k. Yields unusually fluid melt
- l. Received late in program
- m. Poor friction characteristics, not tested further

TABLE 1 - Continued

Preliminary Candidate Screening - Peel Strength (1/2" Width Sample)

Sample I.D. Number	Adhesive	Laminate Material	Adhesive Failure (g/in.) Range	Water Immersed at RT for 48 hrs (g/in.)
22008-5	Bostick 7178	Nylon	0	h.
		Nylon	0	h.
		Mylar	0	h.
		Mylar	0	h.
22008-6	Serfene 181	Nylon	500-550	h.
		Mylar	0	h.
22008-7	Serfene 2060 ^e	Nylon	1000	300-400 ^e
		Nylon	600-800 (Nylon tore)	(Nylon tore)
		Mylar	(Mylar tore)	>1000
		Mylar	1000	0-100
				100-220
22008-8	Elvax 4310	Nylon	800-1900	m.
		Nylon	1600-1900	m.
		Mylar	220-400	m.
		Mylar	240-400	m.
22009-1	Macromelt 6301 ^j	Nylon	400-1200	l.
		Nylon	800-1300	l.
		Mylar	0	l.
		Mylar	0	l.
22009-2	Macromelt 6071 ^j	Nylon	500-800	l.
		Nylon	400-800	l.
		Mylar	400-500	l.
		Mylar	300-700	l.
22009-3	Macromelt 6030	Nylon	0	h.
		Nylon	0	h.
		Mylar	0	h.
		Mylar	0	h.
22009-4	Uni-Rez 2655	Nylon	800-1600	60-450
		Nylon	800-1300	20-360

- a. Molded and press-laminated 330° F
- b. Aged less than 7 days
- c. Molding and laminating at 275° F
- d. Molding and laminating at 300° F
- e. Brittle when wet
- f. Number of experiments as shown in table
- g. Peel values generally oscillate during test
- h. Test eliminated due to substandard dry peel
- i. Grams peel/inch width
- j. Fluid melt
- k. Yields unusually fluid melt
- l. Received late in program
- m. Poor friction characteristics, not tested further

Test laminates were prepared as follows:

First, free-standing sheets of the hot melt coating candidates were prepared as discussed on page 3, using either compression molding or casting of water dispersions.

These hot melt films were then used to prepare nylon/hot melt/Gore-Tex/hot melt/nylon and polyester/hot melt/Gore-Tex/hot melt/polyester peel test laminates. These laminates were prepared at 350 degrees F using a roll with a 10-pound weight. Higher pressures were avoided because they result in adhesive penetration of the Gore-Tex porosity, thereby yielding falsely high peel strength results. Samples were again cut into 1/2-inch-wide strips for peel testing.

Peel strength was determined with the peel force measured at a 90-degree angle to the plane of the sample. Results were reported as the average force in grams per unit width of test laminate. Samples were tested dry and after immersion in water at room temperature for 48 hours. The results appear in Table 2.

Following the wet Gore-Tex versus adhesive peel strength testing, it was sometimes possible to initiate separation between the cover film and the adhesive. This peel strength is also reported in Table 2, last column. Where separations could not be initiated, peel strength is tabulated as "high". This wet peel data may be more significant than the wet peel data of Table 1 since the Table 2 lamination was effected at a lower pressure, more representative of what would be encountered in the end-use.

C. Coated Thread - Laboratory Scale

1. Adhesive Formulations

The remainder of the development work was devoted to application of promising coatings to nylon thread and evaluation of the effects of the coatings on sewability and hydrostatic resistance of sewn and sealed seams. With the exception of Michem 4990, which is a water dispersion, the coating resins were formulated with appropriate solvents as well as, in some cases, a chemical blowing agent. The blowing agent created a closed cell foam which when expanded would aid in sealing the holes with minimal quantities of adhesive melt. The formulations appear in Table 3.

2. Initial Thread Coating

For the initial work, soft finish nylon thread³ was Soxhlet extracted for 24 hours to remove any silicone oil lubricants, or other agents, used to treat the thread.

TABLE 2

Gore-Tex Laminations - Peel Strength (1/2" Width Samples)

Samples I.D. Number	Laminate System	Gore-Tex vs Adhesive Dry ^{a.} (g/in.)	Gore-Tex vs Adhesive Dry ^{b.} (g/in.)	Gore-Tex vs Adhesive Wet ^{c.} (g/in.)	Adhesives vs Cover Film Only Wet ^{d.} (g/in.)
22010-1	Nylon-8061-Gore-Tex-8061-Nylon	420-440 380-520	380	190-400	240-400 180-300
22010-2	Mylar-87061-Gore-Tex-8061 Mylar	500-620 440-980	500	300-340	0 0
22010-3	Nylon-Hytrel 4056-Gore-Tex- Hytrel 4056-Nylon	160-300 350-370	360	180-220	20-40 40-80
22010-4	Nylon-Nucrel 925-Gore-Tex- Nucrel 925-Nylon	260-520 280-500	420	240-430	140 170-200
22010-5	Nylon-Michem 4990-Gore-Tex- Michem 4990-Nylon	330-440 420-460	460	240-460	> 420 > 460
22010-6	Nylon-Estane 5715-Gore-Tex- Estane 5715-Nylon	220-400 420-520	450	220-260	> 400 > 520
22010-7	Nylon-R960-Gore-Tex- R960-Nylon	- 0 - 0	e. e.	e. e.	~ 0 ~ 0
22010-8	Mylar-Estane 5715-Gore-Tex- Estane 5715-Mylar	240-300 220-240	300	220-360	100 ~ 0
22010-9	Mylar-Vitel 4709A-Gore-Tex- Vitel 4709A-Mylar	470 640-800	460	420	high ^{b.} high
22010-10	Mylar-Hytrel 4056-Gore-Tex- Hytrel 4056-Mylar	0-20 0-60	e. e.	e. e.	200-400 300-700
22010-11	Mylar-Serfene 2060-Gore-Tex- Serfene 2060-Mylar	0 0	e. e.	e. e.	40-60 60-100
22010-12	Nylon-Serfene 2060-Gore-Tex- Serfene 2060-Nylon	0-100 60-120	e. e.	e. e.	120-220 140-220
22010-13	Nylon-TPX 11-808-Gore-Tex- TPX11-808-Nylon	770-800 400-480	480	330-370	high ^{b.} high
22010-14	Nylon-Elvax 4310-Gore-Tex- Elvax 4310-Nylon	300-380 300-330	400	320-300	high ^{b.} high
22010-15	Nylon-Macromelt 6071-Gore-Tex- Macromelt 6071-Nylon	220-260 140-260	120	e.	high ^{b.} high
22010-16	Nylon-Macromelt 6301-Gore-Tex- Macromelt 6301-Nylon	300-360 290-380	330-400	340-360	high ^{b.} high

a. Sample strips #1 and #2 (duplicate)

b. Cannot initiate failure between cover
film and adhesive

c. Sample strip #3

d. Peel values generally oscillate during test

e. Test eliminated due to substandard dry peel

f. Grams peel/inch width

TABLE 3

Thread Coating Formulations

Sample I.D. Number	Adhesive	Formulation
22014-1 ^a	(Not adhesive) Celogen OT (Blowing Agent)	48.8 gms Celogen OT, 0.18 gms Tamol 850, 37.3 gms H ₂ O D.I., 0.10 gms R&H CF-10 (ball mill for 24 hrs)
22014-2	TPX 11-808	7 gms TPX 11-808, 11 gms IPA (isopropyl alcohol), 10 gms Toluene (dissolve at 80°C - capped)
22014-4	Elvamide 8061	7 gms Elvamide 8061, 10.5 gms methanol, 10.5 gms methylene chloride (dissolve 40°C - capped)
22016-1	TPX 11-808 & Blowing Agent	7 gms TPX 11-808, 0.14 gms Celogen OT, 11 gms IPA, 0.07 gms Aerosol OT 100%, 10 gms Toluene
22016-2	Elvamide 8061 & Blowing Agent	7 gms, Elvamide 8061, 0.14 gms Celogen OT, 10.5 gms methanol, 0.07 gms Aerosol OT 100%, 10.5 gms methylene chloride
22016-3	Michem 4990 & Blowing Agent	28.0 gms Michem 4990, 0.565 gms #22014-1, 0.098 gms Aerosol OT 100%
22016-4	Estane 5715	20 gms Estane 5715, 80 gms MEK (methyl ethyl ketone)
22016-5	Estane 5715 & Blowing Agent	20 gms Estane 5715, 0.4 gms Celogen OT, 80 gms MEK, 0.2 gms Aerosol OT 100%
22016-6	Macromelt 6071	28 gms Macromelt 6071, 44 gms IPA, 41 gms Toluene
22016-7	Macromelt 6301	28 gms Macromelt 6301, 44 gms IPA, 40 gms Toluene
22021-1	Macromelt 6301 & Blowing Agent	56.0 gms formulation #22016-7, 0.28 gms Celogen OT, 0.14 gms Aerosol OT 100%
22021-2	Macromelt 6071 & Blowing Agent	56.0 gms formulation #22016-6, 0.28 gms Celogen OT, 0.14 gms Aerosol OT 100%

a. This formulation is a water dispersion of the blowing agent for
use in #22016-3

Three-foot lengths of thread were coated on a laboratory scale for initial screening. The thread was pulled through an adhesive solution and up through a 22-mil die to remove excess adhesive. The thread was initially dried at room temperature for 20 minutes and oven dried at 110°C for 20 minutes. The thread was then weighed for coating add-on. To achieve a heavier add-on, the dry thread was recoated a second or third time. Because the coating die diameter was constant, as the thread width increased the coating weight per application generally decreased.

3. Initial Hand Sewing

Adhesives evaluated for initial hand sewing were selected (Table 3) from the results (Tables 1 and 2) of peel strength versus substrates. Candidate resins were not only chosen for their adhesive properties, but also for their ability to flow out at 350°F. Promising resins were allowed to melt on a 350°F-/177°C hot plate and resins with good flow out/low melt viscosity were selected. The resins were also screened with the use of a blowing agent in the formulation. The blowing agent creates a foam which helps seal the holes with minimal quantities of adhesive melt (Table 3).

Two-inch-diameter circles were cut from nylon Taslan/Gore-Tex/tricot knit cloth laminate material⁴ and were sewn with the coated thread. Initial sewing was done with a single thread using a hand sewing needle, three stitches per circle, as illustrated by Figure 1. Subsequent hand sewing included: double thread i.e. Figure 2 and quadruple thread, i.e., Figure 3. Some hand stitching was undertaken with a sewing machine needle, leaving a double thread in the hole, i.e., Figure 4. The purpose of two and four threads passing through the needle hole is to simulate a sewing machine stitch wherein two to four threads will pass through the hole (see Figure 5, page 13). Note that six needle holes see the hydraulic pressure, and leakage will occur through the least well-sealed area.

The sewn circles were then placed in a 350°C oven for 30 minutes to allow the hot melt adhesive to melt, wick out, and seal the holes. The formulations containing a blowing agent were placed in the oven for 12 minutes. Previous studies at Springborn Laboratories⁵ have shown that a longer time causes the foam that forms to collapse after approximately 12 minutes.

The samples were then subjected to water under pressure using a laboratory pressure filter. The samples were mounted in the pressure filter bracket and the device filled with water and subjected to pressure. Pressure was applied at a rate of 1 psi/second. The pressure is recorded when water begins to drip from the funnel exit. This device was only used for initial screening. Results are presented in Tables 4, 5, 6, 7 and summarized in Table 8.



Cross Section of Single Thread in Needle Hole

Figure 1: Test Specimen, Three Stitch, Single Thread



Cross Section of Double Thread in Needle Hole

Figure 2: Test Specimen, Three Stitch, Double Thread



Cross Section of Quad Thread in Needle Hole

Figure 3: Test Specimen, Three Stitch, Quadruple Thread

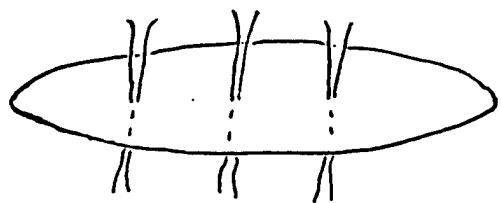
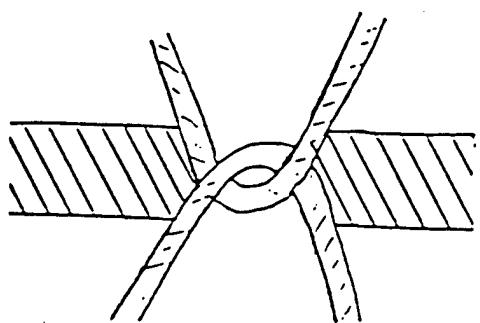


Figure 4: Test Specimen, Sewing Machine Needle,
Three Stitch, Double Thread



Cross Section of Sewing
Machine Stitch

Figure 5: Sewing Machine Stitch, Loop Within
Needle Hole (Type 301)

TABLE 4

Preliminary Screening - Pressure Filter Test
(Single Thread)^b.

Sample I.D. Number	Adhesive	Wt. of 1' Thread (gms)	Wt. Coating & Thread (gms)	% Weight Add-on	# of Dips	Pressure ^a . Test (psi)
Control	None	0.0235	-	-	-	Gravity Leak < 1
22015-1A	TPX 11-808	0.0235	0.0248	5.2	1	10
22015-1B	25% Solids Formulation.	0.0235	0.0257	8.5	2	5
22015-1C	# 22014-2	0.0235	0.0274	14.2	3	9
22017-4A	TPX 11-808 W/Blowing	0.0235	0.0281	16.37	1	12
22017-4B	Agent Formula.	0.0235	0.0319	26.33	2	14
22017-4C	#22016-1	0.0235	0.0351	33.05	3	16
22015-3A	8061	0.0235	0.0310	24.2	1	10
22015-3B	Formulation	0.0235	0.0368	36.14	2	10
22015-3C	#22014-4	0.0235	0.0400	41.25	3	10
22017-5A	8061 w/Blowing	0.0235	0.0300	21.66	1	14
22017-5B	Agent Formula.	0.0235	0.0358	34.35	2	10
22017-5C	#22016-2	0.0235	0.0429	42.22	3	10
22017-6A	Macromelt 6071	0.0235	0.0292	19.52	1	11
22017-6B	Formulation	0.0196	0.0279	28.42	2	13
22017-6C	#22016-6	0.0235	0.0379	37.99	3	13
22022-2A	Macromelt 6071	0.0235	0.0295	20.33	1	13
22022-2B	w/Blowing	0.0235	0.0376	37.5	2	13
22022-2C	Agent Formula.	0.0235	0.0388	39.43	3	15
22017-7A	Macromelt 6301	0.0235	0.0292	19.52	1	11
22017-7B	Formulation	0.0235	0.0337	30.26	2	12
22017-7C	#22016-7	0.0235	0.0376	37.5	3	12
22015-5A	Michem Prime	0.0235	0.0333	29.4	1	8
22015-5B	4990 Neat	0.0235	0.0355	33.8	2	8
22015-5C		0.0235	0.0400	41.25	3	10
22017-3A	Michem Prime	0.0235	0.0300	21.66	1	10.5
22017-3B	4990 w/Blowing	0.0235	0.0331	29.03	2	10
22017-3C	Agent Formula.	0.0235	0.0391	39.89	3	12
22017-1A	Estane 5715	0.0235	0.0280	16.07	1	0
22017-1B	Formulation	0.0235	0.0329	28.57	2	7.5
22017-1C	#22016-4	0.0235	0.0371	36.65	3	8
22017-2A	Estane 5715	0.0235	0.0279	15.77	1	11
22017-2B	w/Blowing Agent	0.0235	0.0333	29.42	2	10
22017-2C	Formu.#22016-5	0.0235	0.0366	35.79	3	10

a. Six threaded needle holes per test; number of experiments as shown in table

b. As in Figure 1

TABLE 5

Pressure Filter Test - Double Thread^{c.}

Sample I.D. Number	Adhesive	Wt. of 1' Thread (gms)	Wt of Coating & Thread (gms)	Percent Wt. Add-on	# of Dips	Pressure Test a. (psig) b.
22024-1	TPX 11-808 Formulation #22014-2, Thread #22015-1C	0.0235	0.0274	14.2	3	14
22022-3	TPX 11-808 w/ Blowing Agent Formulation #22016-1, Thread # 22017-4C	0.0235	0.0351	33.05	3	18
22022-4	Macromelt 6071 Formulation #22016-6, Thread #22017-6C	0.0235	0.0379	37.99	3	14
22022-6	Macromelt 6071 w/Blowing Agent, Formulation #22021-2, Thread #22022-2C	0.0235	0.0388	39.43	3	27
22022-5	Macromelt 6301 Formulation #22016-7, Thread #22017-7C	0.0235	0.0376	37.5	3	23
22024-3	Michem Prime 4990 Neat, Thread #22015-5C	0.0235	0.0400	41.25	3	13
22024-5	Michem Prime 4990 w/ Blowing Agent Formulation #22016-3, Thread #22017-3C	0.0235	0.0391	39.89	3	18
22024-4	Estane 5715 w/Blowing Agent Formulation #22016-5, Thread #22017-2C	0.0235	0.0366	35.79	3	14
22024-6	8061 w/ Blowing Agent Formulation #22016-2, Thread #32017-5C	0.0235	0.0429	42.22	3	12

a. Heat 30 minutes 117°C before testing;
12 minutes heating for foam formulations

b. Six threaded needle holes per test; number of experiments as shown in table

c. As in Figure 2

TABLE 6

Pressure Filter Test - Double Thread
(Sewing Machine Needle Puncture)^a.

Sample I.D. Number	Adhesive	Wt. of 1' Thread (gms)	Wt. of Coating & Thread (gms)	Percent Wt. Add-on	Number of Dips	Pressure Test (psi) c.
22025-1A	Macromelt 6301 Formulation #22016-7	0.0235	0.0387	39.2	3	14
22025-2A	Macromelt 6071 w/ Blowing Agent, Formulation #22021-1	0.0235	0.0364	35.4	3	14 ^b
22025-3A	Macromelt 6071 Formulation #22016-6	0.0235	0.0375	37.3	3	15
22025-4A	TPX 11-808 w/ Blowing Agent Formulation #22016-1	0.0235	0.0375	37.3	3	17 ^b
22025-5A	Michem Prime 4990 (neat)	0.0235	0.0432	45.6	3	25
22025-6A	Michem Prime 4990 w/ Blowing Agent Formulation #22016-3	0.0235	0.0412	42.9	3	>40

a. Hand sewn as in Figure 4

b. Possible foam collapse (in oven over 12 minutes)

c. Six threaded needle holes per test; number of experiments as shown in table

TABLE 7
Pressure Filter Test - Quadruple Thread^{c.}

Sample I.D. Number	Adhesive	Wt. of 1' Thread (gms)	Wt. of Coating & Thread (gms)	% Weight Add-on	Number of Dips	Pressure Test ^{b.} (psi)
22025-1B	Macromelt 6301 Formulation #22016-7	0.0235	0.0380	38.15	3	14
22025-2B	Macromelt 6071 w/Blowing Agent Formulation #22021-1	0.0235	0.0368	36.14	3	8 ^{a.}
22025-3B	Macromelt 6071 Formulation #22016-6	0.0235	0.0374	37.1	3	32
22025-4B	TPX 11-808 w/Blowing Agent Formulation #22016-1	0.0235	0.0375	37.3	3	4 ^{a.}
22025-5B	Michem Prime 4990 (Neat)	0.0235	0.0435	45.9	3	> 45
22025-6B	Michem Prime 4990 w/Blowing Agent Formulation #22016-3	0.0235	0.0423	44.4	3	38

- a. Possible Foam Collapse
(in oven over 12 minutes)
- b. Six threaded needle holes per test;
number of experiments as shown in table
- c. As in Figure 3

TABLE 8

Summary, Pressure Filter Tests^{c.}

Adhesive	Sample I.D. Number	Hydrostatic Pressure Resistance (psi) ^{b.}				% Weight Add-on per Thread
		Single Thread ^{d.}	Double Thread ^{e.}	Double Thread Machine Needle ^{f.}	Quadruple Thread ^{g.}	
TPX 11-808 w/ Blowing Agent, Formulation #22016-3	22017-4C	16	-	-	-	33.05
	22022-3	-	18	-	-	33.05
	22025-4A	-	-	17 ^{a.}	-	37.3
	22025-4B	-	-	-	4 ^{a.}	37.3
Macromelt 6301 Formulation #22016-7	22017-7C	12	-	-	-	37.5
	22022-5	-	23	-	-	37.5
	22025-1A	-	-	14	-	39.2
	22025-1B	-	-	-	14	38.15
Macromelt 6071 w/Blowing Agent Formulation #22021-2	22022-2C	15	-	-	-	39.43
	22022-6	-	27	-	-	39.43
	22025-2A	-	-	14 ^{a.}	-	35.4
	22025-2B	-	-	-	8 ^{a.}	36.14
Macromelt 6071 Formulation #22016-6	22017-6C	13	-	-	-	19.52
	22022-4	-	14	-	-	37.99
	22025-3A	-	-	15	-	37.3
	22025-3B	-	-	-	32	37.1
Michem Prime 4990 (Neat)	22015-5C	10	-	-	-	41.25
	22024-3	-	13	-	-	41.25
	22025-5A	-	-	25	-	46.6
	22025-5B	-	-	-	> 45	45.9
Michem Prime 4990 w/Blowing Agent Formulation #22016-3	22017-3C	12	-	-	-	39.89
	22024-5	-	18	-	-	39.89
	22025-6A	-	-	> 45	-	42.9
	22025-6B	-	-	-	38	44.4

- a. Possible foam collapse (in oven over 12 minutes)
- b. Six threaded needle holes per test; number of experiments as shown in table
- c. Data taken from Tables 4,5,6 and 7
- d. As in Figure 1
- e. As in Figure 2
- f. As in Figure 4
- g. As in Figure 3

D. Evaluation of Volatile Yarn Lubricants

Friction between the coating and the metal needle may cause heating of the needle resulting in a softening of the coating. This in turn would impede the sewing process. It was thought that the friction caused by the coating could be reduced by the use of a volatile lubricant.

Preliminary friction measurements were carried out using an 860-g steel bar attached to a spring scale. The film to be screened was placed on a glass strip with rounded side edges, and the metal weight was pulled along its surface. Friction was measured in grams of force required to move the steel bar across the film. The results appear in Table 9.

Lubricants were evaluated in a friction test using films of Michem 4990[®] Prime. Table 10 shows the comparison of the dry film with a lubricated film.

E. Hydrostatic Pressure Resistance

Five of the most promising candidate formulations were coated onto thread and evaluated for hydrostatic resistance per Test Method 5512.⁶ Candidate resins were chosen for their adhesion to nylon and/or Mylar and Gore-Tex, as well as the results of the initial pressure filter.

The thread was coated (3 dips) and hand-sewn into the cloth as double thread. The samples were then placed in a 350°F oven for 30 minutes except for the blowing agent formulations which only remained at 350°F for 12 minutes. Results are presented in Table 11.

*-----

* This test procedure was carried out by U.S. Testing Co., Inc.

TABLE 9

Friction Test - Adhesive vs Metal

<u>Adhesive</u>	<u>Friction - Grams</u> ^{a.}
Serfene 2060	160, 170, 170
Vitel 4709-A	170, 170, 180
Elvamide 8061	180, 190, 180
TPX 11-808	180, 180, 200
Estane 5715	180, 200, 190
R-960	280, 250, 260
Nucrel 925	310, 300, 320
Michem 4990	350, 370, 400
Elvax 4310	550, 500, 550
Hytrel 4056	600, 620, 650

a. Triplicate determinations

TABLE 10

Michem Prime 4990
Friction Test w/Various Lubricants

<u>Lubricant</u>	<u>Friction - Grams</u>	
	<u>Dry (Control)</u>	<u>w/ Lubricant c.</u>
3M FC-70 (Fluorocarbon)	350	50 - 150
Union Carbide Volatile Silicone 7158	300	175 - 300 (200 Avg)
2% Polyvinylpyrrolidone (PVP) a. K-90 GAF	375	175 - 300 (200 Avg)
2% PVP + 1% Union Carbide Silwet L-77 a.	400	100 - 300 (200 Avg)
2% PVP + 1% Air Products Surfynol 104 a.b.	400	100 - 200
1.5% PVP + 0.25 Surfynol 104 a.	400	200 - 300 (250 Avg)

- a. Aqueous solution
- b. Not entirely soluble
- c. Duplicate determinations

TABLE 11
Hydrostatic Pressure Resistance

Sample I.D. #	Adhesive (Formulation #)	* Wt. Add-on	Hydrostatic Pressure Resistance (psi) ^a		
			Individual	Average	Median
22029-1A	Control	--	74	93	98
22029-1B	(Unsewn circles)		100		
22029-1C			104		
22029-1D	(Cloth as received)		88		
22029-1E			98		
22029-2A	Uncoated	--	0	0	0
22029-2B	Thread		0		
22029-2C			0		
22029-2D			0		
22029-2E			0		
22027-1A	Michem	44.7	28	57	80
22027-1B	4990		7		
22027-1C	Neat		84		
22027-1D	#22026-1		86		
22027-1E			80		
22027-2A	Michem	41.3	10	28	10
22027-2B	4990		30		
22027-2C	w/blowing		10		
22027-2D	agent		78		
22027-2E	#22026-2		10		
22027-3A	Macromelt	49.4	6	10	10
22027-3B	6301		10		
22027-3C	#22026-3		10		
22027-3D			16		
22027-3E			10		
22027-4A	Macromelt	53.6	0	16	8
22027-4B	6301		64		
22027-4C	w/blowing		0		
22027-4D	agent		8		
22027-4E	#22026-8		8		
22027-5A	Uni-Rez	37.9	0	4	6
22027-5B	2655		6		
22027-5C	#22026-7		0		
22027-5D			6		
22027-5E			6		

a. Double thread, three stitches in each of five circles
Fed. Std. 191A, Method 5512

III. DISCUSSION OF RESULTS

A. Initial Screening

1. Peel Strength Adhesion of the Hot Melt Coatings to Nylon and Polyester

The peel strength results of the initial screening, Table 1, indicated that the hot melts with the greatest adhesion to nylon and polyester were as follows:

<u>For Nylon</u>	<u>For Polyester</u>
Estane 5701 ®	Vitel 4709A ®
Estane 5715 ®	Estane 5715 ®
Dow EAA 459 ®	Hytrel 4056 ®
TPX 11-808 ®	Serfene 2060 ®
Elvamide 8061 ®	Elvax 4310 ®
Michem 4990 ®	Macromelt 6071 ®
Nucrel 699 ®	
Nucrel 925 ®	
Serfene 2060 ®	
Serfene 181 ®	
Elvax 4310 ®	
Macromelt 6301 ®	
Macromelt 6071 ®	
Unirez 2655 ®	
NeoRez R960	

To shorten the candidate list, those adhesives with relatively poor peel strength were dropped, for example, since the Nucrels are all chemically similar and Nucrel 925 provided better peel strength than Nucrel 699, the latter was dropped. Similarly, Serfene 2060 appeared to be superior to Serfene 181 and Estane 5715 was more promising than Estane 5701.

Hot melt laminates showing good peel strength were soaked 48 hours in water and retested, last column (Tables 1 and 2). Table 1 wet peel strength values, with few exceptions, were higher than Table 2 wet laminate values. This can be attributed to the higher lamination pressure used for the initial (Table 1) lamination.

Promising hot melts at this point included:

<u>For Nylon</u>	<u>For Polyester</u>
Estane 5715	Estane 5715
Elvamide 8061	Hytrel 4056
Michem 4990	Vitel 4709A
Nucrel 925	
Uni-Rez 2655	
TPX 11-808	
Elvax 4310	
Macromelt 6301	
Macromelt 6071	

2. Peel Strength Adhesion of the Hot Melt Coating to Gore-Tex

Peel strength data for Gore-Tex laminates, dry and wet are presented in Table 2. Additional data on the peel strength of laminates after water immersion is also included in Table 2, in last column. On the basis of these results, the more promising adhesives at this point were:

<u>Nylon to Gore-Tex</u>	<u>Polyester to Gore-Tex</u>
Elvamide 8061	Vitel 4709A
Nucrel 925	
Michem 4990	
Estane 5715	
TPX 11-808	
Elvax 4310	
Macromelt 6071	
Macromelt 6301	

3. Evaluation of Coated Thread

To reduce the number of candidates, Nucrel and Elvax were dropped at this time on the basis of their poor friction characteristics (Table 9). Michem 4990 has relatively poor friction characteristics, but was retained as a candidate because of its very fluid melt characteristics.

The coated thread described in Section II-C of this report and in Table 3 was sewn in place with a hand stitching needle and heated to melt the coating. Sewn and sealed samples were subjected to hydrostatic testing.

Table 4 shows the results of samples sewn with a single thread at different coating weight add-ons. The control was a piece of cloth sewn with uncoated thread in the same manner as the samples using the coated thread. The control leaked water simply by gravity.

For most of the candidate resins, the results show an increase in pressure resistance with the use of a chemical blowing agent. The blowing agent was used to create a closed cell foam from the coating.

One would expect better sealing properties with increased coating weight, but this was not true for four of the experiments. For the single thread experiments, reported in Table 4, coating weight of over 20 to 28-percent resulted in little or no improvement in hydrostatic resistance.

Table 5 reports hydrostatic resistance results of the candidate coatings using a double thread, i.e. Figure 2. The results are substantially improved over those using a single thread.

It was thought that the hand-sewing needle technique was more damaging to the cloth than would be the case with a sewing machine needle. Therefore, stitches were made in the cloth with a sewing machine needle (size 16, 0.040-inch diameter) puncture. The thread was then drawn through this hole, Figure 4. The hydrostatic resistance results (Table 6) improved with some but not all of the samples over those using the hand sewing needle.

The samples were also hand-sewn using four threads passing through the hole to simulate a sewing machine stitch wherein the loop is within the needle hole (Figure 5) rather than above or below the needle hole. Table 7 presents pressure filter results with four threads per needle hole.

Table 8 shows a comparison of the results in Tables 4 to 7. Note that the "A" designation means 1 dip, "B" is 2 dips, and "C" or no letter is 3 dips of the coating. Using thread with Macromelt 6301 and Michem 4990, neat (no foaming agent), the quad (quadruple) thread provided the best hydrostatic resistance. One might expect that four threads would "fill" the needle hole better than two threads, i.e. see Figure 3 cross section versus Figure 4 cross section.

Although a double thread should be more difficult to seal against high pressure than a quadruple thread, Michem 4990 (22025-5A and 22025-6A, Table 8) showed good initial promise in sealing sewing machine needle puncture holes.

A dilute methyl violet/fluorocarbon detergent dye solution was used as a test vehicle to determine where the leaks had originated. Following the pressure filter test, the samples were dried and the stitches on the camouflage side of the sewn samples were painted with the dye solution. The reverse side of the sample was observed for penetration of the dye around the needle holes, or the dye penetrating the coating and wicking up via the inside of the thread bundle.

The TPX 11-808 samples showed penetration of the dye around the holes, but not discoloring the coated thread. The Elvamide 8061 did show discoloration through the braid of the thread, indicating penetration of the coating. The Michem 4990 and the Macromelt 6071 never showed penetration of any kind. The dye did not penetrate the intact nylon Taslan/Gore-Tex/tricot knit cloth laminate.

Lengths of thread coated with final candidate hot melts were sewn into barrier cloth, heat-fused and subjected to hydrostatic resistance per Test Method 5512, Table 11. Michem 4990 yielded the most consistent pressure resistance and the highest leak pressures. Hydrostatic results for coated thread using foaming agent were erratic, ranging from good to poor.

4. Pilot Plant Coated Thread/Machine Sewing

The Pilot plant-coated thread was coated as received with Michem 4990. No particular difficulty was encountered although, to obtain a well-fused coating, a significant amount of heat was needed in the drying section. The coated thread gave no handling problem when used on a small sewing machine operating at maximum speed (approximate 10 feet/minute).

Heat-fused machine-stitched barrier cloth, however, leaked at 12-14 psi (laboratory pressure filter). The solvent-extracted laboratory-coated white thread, on the other hand, when hand sewn and sealed gave stitches (Figure 5) which leaked at >35 psi. Thus, it would appear that special lots of thread without sizing will have to be obtained and utilized in the future for pilot plant coating.

5. Volatile Yarn Lubricants

The data presented in Table 9 indicates that some of the better hot melt adhesives (e.g. Michem 4990) have rather high friction characteristics against steel. This could result in softening/melting problems at very high sewing speeds, although the newer friction-resistant needles and the use of air cooled needles may prevent such problems. In the event friction problems arise, some precautionary work was undertaken to identify volatile lubricants that would enable high sewing speeds, but would evaporate before interfering with the bonding step.

The Table 10 results indicate that 3M's volatile fluorocarbon is by far the most efficient lubricant. Less expensive lubricants such as silicone, or dilute aqueous polyvinyl pyrrolidone, appear to be helpful but are inferior to the fluorocarbon.

B. Most Promising Candidate

Michelman Chemical's Michem Prime 4990 is an aqueous alkaline dispersion of Dow Chemical's Primacor Resin (EAA - ethylene acrylic acid). It was chosen for its performance in overall initial screening and from final hydrostatic resistance results.

1. Thread Coating

A pilot plant thread coating apparatus was assembled to coat the larger amounts of thread needed for machine sewing. A green nylon thread similar to the white soft finish thread was obtained in larger quantities and was used as received. (Solvent washing tended to extract much of the color). A drawing of the apparatus is presented in Figure 6. The thread was routed through the adhesive solution and pulled up through a 22-mil die to remove the excess adhesion. The thread was then passed through an "air-circulating oven" at ~350°F to dry, followed by two fusing steps with infrared lamps, and the thread was then finally wound on a take-up spool. This process was repeated with previously coated thread to obtain higher weight add-ons.

There were initial problems with the coating on the thread being easily removed, (e.g., could be scraped off with a sharp object). Improvements in the drying and fusing process solved this problem with minimal difficulty. The oven temperature gradient was raised and the thread was moved closer to the IR lamps.

2. Machine Sewing

A standard 301 Lockstitch Singer sewing machine was used to test the coated thread for sewability. This is a commercially available household type machine, equipped with a size 18 (0.044-inch-diameter) needle.

Double-coated and triple-coated threads were evaluated as well as a control (uncoated thread) for sewability. The sewability was measured by recording sewing speed on a one-foot section of material. The uncoated thread stitched (full speed) at the rate of 11.5 feet/minute. The double-coated thread stitched almost as fast, 9.8 feet/minute. The triple-coated thread stitched at the rate of 9.7 feet/minute. All coated threads were sewn with the use of FC-70, a 3M volatile fluorocarbon, as a lubricant.

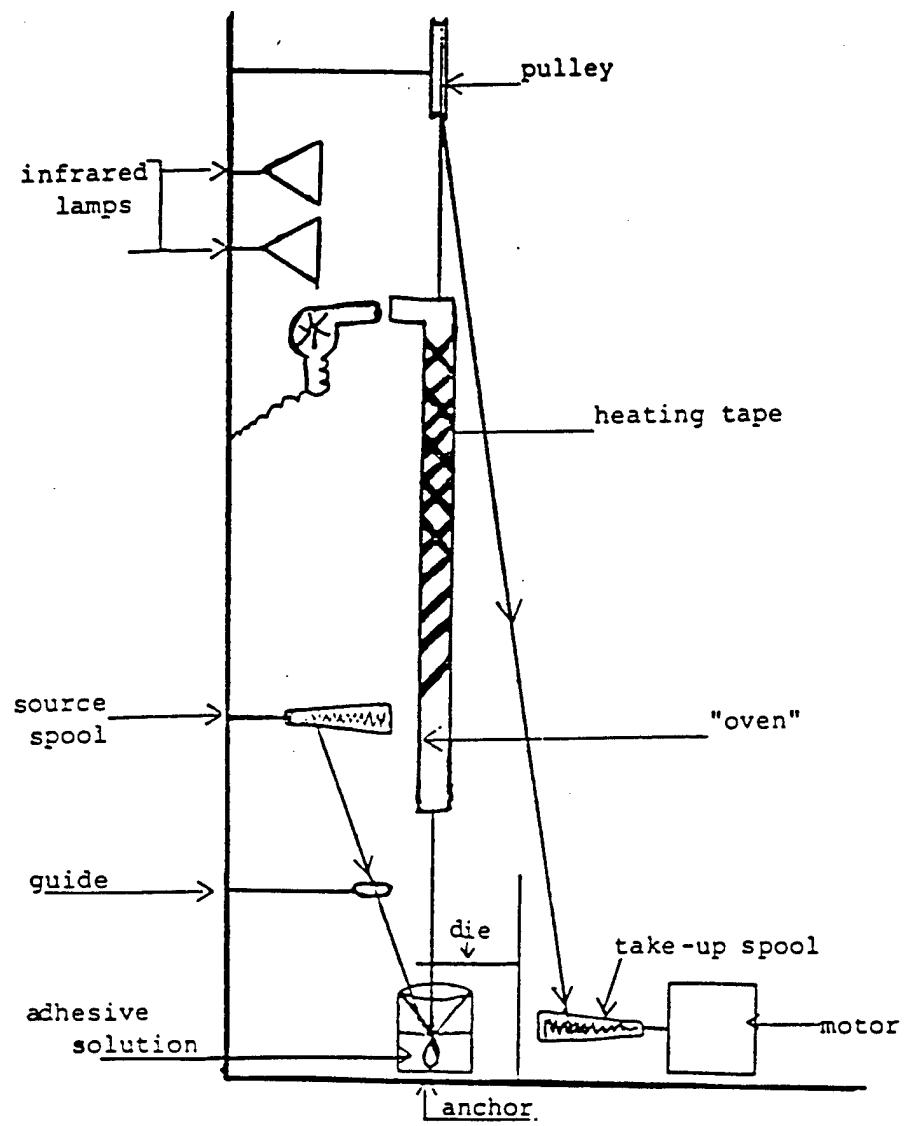


FIGURE 6: Pilot Plant Thread Coater

3. Machine Sewn Samples

Machine sewn samples were prepared using pilot coated green thread, thread which had not been solvent extracted prior to coating. Samples were prepared using thread which had been both double-coated and triple-coated.

In order to compare the effect of sewing process on sealed seam properties, additional samples were also prepared by hand sewing. These samples used a double-coated green thread.

The sewn and sealed samples, which were later subjected to hydrostatic resistance testing, consisted of nylon Taslan/Gore-Tex/tricot knit discs with 6 sewn and sealed stitches. Machine sewn samples were prepared with a standard 301 Lockstitch Singer sewing machine.

The glossy finish on the coated thread became dull after sewing, suggesting a possible loss of some of the coating. The machine sewing created larger needle holes in the fabric than did the hand sewing, which could influence hydrostatic resistance.

Hydrostatic resistance was measured on all samples using a pressure filter. Hydrostatic resistance for both the hand and machine sewn samples was in the range of 12 to 14 psig. This contrasts with the hydrostatic resistance of greater than 35 psig obtained with samples hand sewn and sealed with coated white thread. The white thread, of course, had been solvent extracted prior to coating.

Dye testing was not helpful in determining the leakage path for tested samples.

These results suggest that solvent extraction will be required on all thread prior to coating in order to obtain adequate hydrostatic resistance in the final sewn and sealed seams.

IV. CONCLUSIONS

This Phase I development program successfully demonstrated the concept of a heat sealable sewing thread for use in the outer shell fabric laminate of a chemical protective garment. Specifically, this development effort demonstrated the feasibility of coating a sewing thread with a hot melt resin, using that coated thread to sew a nylon Taslan/Gore-Tex/tricot knit laminate, and then heating the sewn area to produce a seam with hydrostatic resistance of at least 35 psig. The brief 350°F 30-minute heating did not appear to damage the cloth and thread.

Based on this Phase I work, it is feasible to machine sew the coated thread. Volatile fluorocarbon oils have been identified which can be used as thread lubricants to facilitate the machine sewing process.

Successful bonding of nylon thread to Gore-Tex was achieved with a number of hot melts; Elvamide 8061, Nucrel 925, Michem 4990, Estane 5715, TPX 11-808, Elvax 4310, and Macromelts 6301 and 6071. Of the hot melts evaluated for adhesion to polyester and Gore-Tex, the Vitel 4709A showed the most promise.

Michelman/Dow Michem 4990, a waterborne ethylene acrylic acid, was the most promising resin for thread coating. Fabric samples, sewn and sealed with Michem 4990 coated thread, withstood the highest hydrostatic resistance in the most reproducible manner. Using a household sewing machine, thread triple-coated with Michem 4990, sewed with comparable speed to that of the uncoated thread.

V. RECOMMENDATIONS

Sewability

- o The primary consideration for the next phase of this program would be to design a process in which the sewing and heat sealing of the coated thread could be carried out at one work station. The sewing machine station may be adapted in the following ways to incorporate the heat sealing process:
 - . two heated 5/8" Teflon coated opposing rollers,
 - . one heated roller and a Teflon-coated plate,
 - . hot air jets,
 - . ultrasonic horn-driven wheel or 1x5/8-inch surface.
- o In Phase I a lubricant was used to aid the sewing process. It was assumed the friction and heat generated during sewing may cause the coating to soften and become tacky, which would in turn impede the sewing. The lubricant (3M's FC-70) is expensive. The following alternatives should be considered:
 - . evaluate Teflon-coated needles to reduce friction,
 - . evaluate other less expensive lubricants as an alternative lubricant,
 - . test various coated threads without lubrication.
- o The two industrial machines (301, 401) presently used produce two different kinds of stitches.

The 301 gives the conventional stitch that looks the same on the needle side as it does on the bobbin side with the interlocking loop occurring (ideally) within the cloth.

The 401 produces the conventional stitch on top, but the under-side is a series of loops (a greater thread mass). The 401 is also a faster machine than the 301.

Future work involving these two machines should include the following:

- . Compare the sewability of the two machines with the coated thread. There may only be a need to use coated thread on the bottom stitch due to the increased thread mass and therefore, coating mass.
- . Investigate a steam press for sealing the thread holes in Chemical Protective (CP) uniforms.
- . Investigate methods for factory and field repair of leakers. Develop a liquid formulation.

Formulations

The present study (Phase I) has primarily dealt with nylon and Gore-Tex as the substrates of interest. Ideally, an adhesive (coating) with more universal application is needed. Steps to achieve this should include the following:

- o Future formulations would include new materials and reformulations of old materials (for nylon thread) in order to obtain minimum coating weight for foolproof sealing versus maximum coating weight for sewability (first with household, then industrial sewing machines).
- o Investigate coatings for thread to be used with neoprene-coated waterproof fabric.
- o Research coatings for polyester core thread with spun cotton coating.
- o Evaluate coatings for thread used in vinyl coated polyester (e.g., tents).
- o Determine materials to coat Belding Corticelli's "NYMO" Monocord. Meet with their personnel to discuss substituting our Michem 4990 type binder for their present binder in experimental lots.
- o Investigate ultraviolet resistance with and without UV screening agents in the formulation.
- o Examine formulations and materials for bonding to other barrier resins such as solid TPU sheet (e.g., BION II).
- o Detailed work with foaming formulations should, also, be carried out, especially with foam stabilizers; lesser amounts of foaming agent.
- o Alternate source of supply for Michem 4990.
- o Some work will have to be undertaken to establish whether or not unsized thread has to be utilized for thread coating.
- o It will have to be established if sizes can be utilized on thread for Extended Cold Weather Clothing System (ECWCS) or Aircrew Uniform Integrated Battlefield (AUIB) applications.

Testing

The Phase II program should involve extensive testing of the materials and final products along with failure analysis. The following is suggested:

- o Evaluation of coated thread in ECWCS laminates for water repellent use.
- o Evaluation of coated thread in AUIB laminates for CP use.
- o Investigation of laundering, dry cleaning resistance.
- o Determine thread tensile strength before and after coating with Michem 4990.
- o Evaluation of abrasion resistance of the thread with and without Michem 4990 impregnation.
- o Evaluation of tentage with treated thread by cup test.
- o Investigation of dye leaks with a microscope and/or an electron microscope. Analyze failures, i.e., wicking along the inside of the twisted thread, wicking between thread and Gore-Tex, or possible "trap-door" failure of Gore-Tex.
- o Analyze failure of foam formulations.
- o Evaluate wear and tear testing of the hot melt thread sealed clothing, 5 uniforms.
- o Evaluate flammability.
- o Evaluate agent resistance.

This document reports research undertaken in cooperation with the US Army Natick Research, Development and Engineering Center under Contract No. DAAK60-86-C-0089 and has been assigned No. NATICK/TR-87/046 in the series of reports approved for publication.

LIST OF REFERENCES

- (1) Gore-Tex, U.S. Patent No. 4,194,041, "Waterproof Laminate", March 18, 1980.
- (2) G. Byers, W. Holley, C. Mowrey, A. Siniscalchi and M. Walker "Development of Water Resistant Coatings for Chemical Protective Clothing", Springborn Laboratories, Inc., Contract DAAK60-84-C-0052, Natick/TR-86/041L, June 1986 (AD B104 481)
- (3) Federal Specification, V-T-295 "Thread, Nylon", dtd February 3, 1977
- (4) Military Specification, MIL-C-44187 "Cloth, Laminated, Waterproof and Moisture Vapor Permeable" dtd Oct. 10, 1985
- (5) Springborn Laboratories private client work (unpublished).
- (6) Federal Standard 191A, Test Method 5512, "Water Resistance of Coated Cloth; High Range, Hydrostatic Pressure Method" dtd July 20, 1978.

APPENDIX

Hot Melt Materials

APPENDIX

Hot Melt Materials

<u>Materials</u>	<u>Manufacturer</u>	<u>Type Resin</u>
Vitel 4709A	Goodyear	Polyester
Vitel 10,202	Goodyear	Polyester
TPX 11-808	Henkel Corporation	Polyamide
Duraflex 8910	Shell Chemical Co.	Polybutylene
Nucrel 699	DuPont	Ethylene/Methacrylic Acid Copolymer
Nucrel 925	DuPont	Ethylene/Methacrylic Acid Copolymer
EAA 459	Dow	Ethylene/Acrylic Acid
Surlyn 9970	DuPont	Ionomer
Estane 5715	B.F. Goodrich	Polyurethane
Estane 5701	B.F. Goodrich	Polyurethane
Bostik 7376	Bostik	Polyester
Bostik 7178	Bostik	Polyester
Bostik 7155	Bostik	Polyester
Michem 4983 Prime	Michelman/Dow	Ethylene Acrylic Acid Water Dispersion
Michem 4990 Prime	Michelman/Dow	Ethylene Acrylic Acid Water Dispersion ("Primacor")
Elvax 4310	DuPont	Ethylene Vinyl Acetate Copolymer
Adcote 50T4983	Morton Thiokol/Dow	Ethylene Acrylic Acid Water Dispersion

<u>Materials</u>	<u>Manufacturer</u>	<u>Type Resin</u>
Adcote 50T4990	Morton Thiokol/Dow	Ethylene Acrylic Acid Water Dispersion
Hytrel 4056	DuPont	Polyester
NeoRez R-960	Polyvinyl Chem	Aliphatic Urethane Dispersion
Serfene 2060	Morton Thiokol	Vinylidene Chloride Dispersion
Elvamide 8023	DuPont	Polyamide
Elvamide 8061	DuPont	Polyamide
Elvamide 8063	DuPont	Polyamide
Serfene 181	Morton Thiokol	Vinylidene Chloride Dispersion
Macromelt 6071	Henkel Corporation	Polyamide
Macromelt 6301	Henkel Corporation	Polyamide
Uni-Rez 2623	Union Camp	Polyamide
Uni-Rez 2620	Union Camp	Polymide
Uni-Rez 2655	Union Camp	Polyamide
Uni-Rez 2645	Union Camp	Polyamide
Uni-Rez 2665	Union Camp	Polyamide
Versalon XR1165	Henkel Corporation	Polyamide
Versalon 1140	Henkel Corporation	Polyamide
Macromelt 6030	Henkel Corporation	Polyamide